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54 Intumescent flexible polyurethane foam and process of manufacture thereof.

57 Flexible resilient flame-retardant and intumescent polyurethane foam is prepared from a reaction mixture comprising a polyester polyol, an organic polyisocyanate, a blowing agent, a surfactant, a catalyst, a melamine derivative and a flame-retardant; the melamine derivative has one or more hydrogen atoms replaced by a methylol and/or lower alkoxymethyl group, the amount of flame-retardant is 20-60 parts by weight/100 parts by weight of the polyester polyol and hydrated alumina is also included in the reaction mixture. In a preferred embodiment, the hydrated alumina is predispersed in a mixture comprising the melamine derivative, the flame retardant and a low-viscosity linear polyester polyol.

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1 INTUMESCENT FLEXIBLE POLYURETHANE FOAM AND PROCESS OF
2 MANUFACTURE THEREOF
3 DESCRIPTION

4 This invention relates to improvements in flexible
5 resilient polyurethane foams and, in particular, to
6 improvements in the characteristics of such foams when
7 subjected to fire.

8 Flexible resilient polyurethane foams are made by
9 the reaction of polyols and organic polyisocyanates
10 in the presence of one or more blowing agents, one or
11 more surfactants and one or more catalysts. The foams
12 find a variety of uses, such as carpet underlay, textile
13 innerlining, mattresses, pillows, furniture padding,
14 cushions, motor vehicle crash pads and thermal insula-
15 tion. Unless formulated with special additives, poly-
16 urethane foams burn readily when ignited. It is known
17 to add various flame-retardant chemicals to poly-
18 urethane foam-forming reaction mixtures, in particular
19 halogenated esters of phosphorus. This kind of addition
20 results in some improvement in the flammability proper-
21 ties, the extent of burning after ignition by a low
22 intensity source being reduced; also, the foams may
23 even be made self-extinguishing to some degree in small-
24 scale laboratory tests; however, when combustion does
25 occur, the foam melts and may drip flaming embers which
26 may ignite other flammable materials in the vicinity
27 and thus cause the fire to spread. In order to overcome

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1 this problem, other additives have been added to
polyurethane foam-forming reaction mixtures, so as to
render the finished foams intumescent or capable of
producing a char, once they have been ignited. Such
5 foams are less prone to the development of flaming
dripping embers during combustion and produce a char
which can act as a thermal insulating barrier and thus
slow or prevent the spread of the fire.

U.S. Patent Specification 3,681,273 describes the
10 use of a mixture of a nitrogen and phosphorus containing
compound, such as monoammonium phosphate, melamine phos-
phate or urea phosphate, and an N-alkylol-substituted
polyalkylene polyamine as an additive to conventional
flexible polyurethane foam reaction mixtures. The
15 resultant foams are flame-retardant, intumescent and
non-burning.

U.S. Patent Specification 3,803,063 discloses a
flexible polyurethane foam reaction mixture containing
an additive comprising a nitrogen and phosphorus
20 containing compound, such as monoammonium phosphate or
an ammonium phosphate and a polycondensate of poly-
merized unsaturated fatty acids with aliphatic amines.

U.S. Patent Specification 3,897,372 discloses
hydrophilic polyurethane foams having flame-retardancy
25 and reduced smoke density, prepared by reacting a resin
prepolymer, water, alumina hydrate, boric acid and a
melamine derivative. Optionally, the reaction mixture
also contains silica.

In U.S. Patent Specification 4,139,501, flexible
30 polyurethane foams having reduced tendency to form
burning embers when ignited and burned are disclosed,
produced by incorporating into the foam-forming reaction
mixture a melamine derivative wherein one or more hydrogen
atoms have been replaced by hydroxymethyl and/or lower
35 alkoxymethyl groups. A halogenated phosphorus ester can

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1 also be included in the reaction mixture.

U.S. Patent Specification 4,143,029 discloses
dripless fire-retarded polyurethane elastomers comprising
a blend of an elastomer with a hexaalkoxymethylmelamine,
5 a trihydrocarbylcarbonylmelamine and an aromatic bis-
maleimide or aromatic bismaleimide generator.

U.S. Patent Specification 4,162,276 discloses non-
dripping self-extinguishing polyurethane elastomers
comprising a blend of an elastomer with a hexaalkoxy-
10 methylmelamine, an organic halogen-containing compound,
such as a chlorinated paraffin, and a P-substituted
phosphocyclopentane-1-oxide.

U.S. Patent Specification 4,197,373 discloses
polyether polyurethane foams prepared from conventional
15 foam-forming reaction mixtures containing, as a flame-
retardant, a melamine derivative having a 2,2,2-tri-
chloromethyl group, such as N,N'-bis(1-hydroxy-2,2,2-
trichloroethyl)melamine.

→ This invention provides flexible resilient polyester
20 polyurethane foams, which have substantially improved
char-forming, or intumescent, properties, as compared with
prior art polyurethane foams, such as those disclosed in
the above-mentioned U.S. Patent Specification 4,139,501.
The improvement is obtained by adding to a polyester
25 polyurethane foam-forming reaction mixture at least one
melamine derivative and at least one flame-retardant,
as specified below, and also hydrated alumina. The one
or more melamine derivatives are those in which one or
more of the hydrogen atoms of melamine have been replaced
30 by hydroxymethyl and/or lower alkoxymethyl groups. The
amount of melamine derivatives used is generally
substantially greater than the amounts described in U.S.
Patent Specification 4,139,501. The amount of flame-
retardant used is also generally substantially greater
35 than the amounts described in U.S. Patent Specification
4,139,501. Relatively large amounts of hydrated alumina

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1 are used in carrying out the invention and, in order
to incorporate these amounts into the foam-forming
reaction mixture, a premix of pumpable viscosity is
preferably prepared, by dispersing the alumina in a
5 synergistic viscosity modifier composition.

The 'flexible foams disclosed in U.S. Patent
Specification 4,139,501 were a significant step forward
in the art, by virtue of their ability to produce a
char when ignited and to resist the formation of dripping
10 embers when ignited. However, still more stringent
performance requirements have recently been placed upon
flexible urethane foams with respect to resistance to
burning, especially for such uses as institutional
mattresses and upholstery, and more rigorous test
15 methods have been developed to evaluate them. Although
these prior art foams give good results when tested in
accordance with the procedures specified in ASTM D
1692-68 or by a modification of UL94 published by
Underwriters Laboratories (the modification being to
20 measure flame time from the start of ignition rather
than from 60 seconds after ignition) and also provide
adequate flame resistance for many applications, they
have proved inadequate when evaluated by recent more
rigorous test procedures. One example of such a
25 procedure is to expose a vertically-held piece of foam
to the flame of a propane gas blowtorch for times
ranging from 10 seconds to 3 minutes. When tested in
this way, conventional urethane foams which do not
contain flame-retardants, as well as those containing
30 conventional levels of such flame-retardants as halo-
genated phosphate esters, ^{entbunden}ignite and continue to burn
after the blowtorch flame is removed until they are
completely consumed. The char-forming foams made
according to U.S. Patent Specification 4,139,501
35 generally continue to burn for a time after the blow-

1 torch flame is removed, although they resist being
totally consumed and are usually self-extinguishing.
Depending upon the thickness of the test piece,
however, the foam may be severely damaged. By way of
5 example; a test piece having a thickness of 25 mm (1 inch)
or less may have a hole burned completely through it.
In contrast to this, when tested by exposure to a blow-
torch, the intumescent foams of the present invention
cease to burn within 0-10 seconds after removal of the
10 torch flame. There is little damage to the interior of
the foam, due to the heavy char layer which forms.

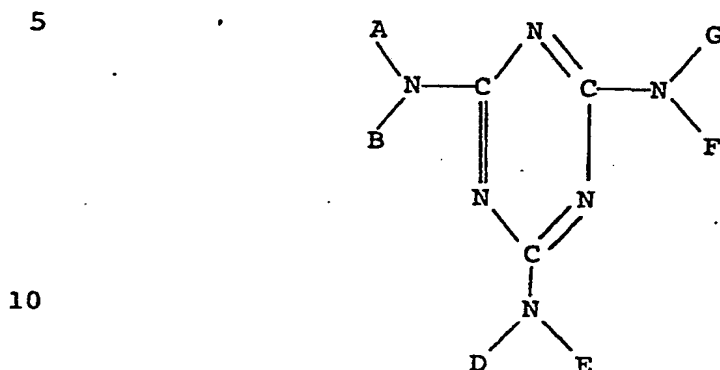
The foams of this invention can be used in most or all
of the applications where conventional flexible foams
have previously been used. Also, these foams can, if
15 desired, be peeled, split or sliced into sheets of any
desired thickness, using techniques well-known in the
art, and the flexible sheets also can be rolled up for
convenient storage and transportation. Like conven-
tional flexible polyurethane foams, the products of the
20 process of this invention can be laminated to textiles
or plastics substrates by conventional methods, such
as flame-lamination or by means of adhesives.

In accordance with the present invention, therefore,
a process is provided for the manufacture of a flexible
25 resilient flame-retardant and intumescent polyurethane
foam from a reaction mixture comprising (a) at least
one polyester polyol, (b) at least one organic poly-
isocyanate, (c) at least one blowing agent, (d) at
least one surfactant, (e) at least one catalyst, (f)
30 at least one melamine derivative and (g) at least one
flame-retardant, wherein:

(1) the or each polyester polyol (a) comprises the
reaction product of at least one polyhydric alcohol
with at least one polycarboxylic acid or anhydride or
35 at least one lactone, the polyester polyol having a
:

1 hydroxyl functionality greater than 2 and a molecular weight of 400 to 5000;

(2) the or each melamine derivative (f) has the formula:



wherein A, B, D, E, F and G are the same or different and each represents a hydrogen atom or a methylol or ROCH_2 group, in which R is an alkyl group having 1 to 4 carbon atoms, with the proviso that at least one of A, B, D, E, F and G is such a methylol or ROCH_2 group; and

(3) the flame-retardant is selected from triesters of phosphoric acid, halogenated triesters of phosphoric acid and halogenated hydrocarbons, and this process is characterised in that the amount of melamine derivative (f) comprises 10 to 30 parts by weight, per 100 parts by weight of polyester polyol (a); that the amount of flame-retardant (g) comprises 20 to 60 parts by weight, per 100 parts by weight of polyester polyol (a); and that the reaction mixture also comprises 50 to 200 parts by weight of (h) hydrated alumina, per 100 parts by weight of polyester polyol (a).

The production of improved polyurethane foams in accordance with this invention is accomplished, therefore, by incorporating into an otherwise conventional polyester polyurethane foam-forming reaction mixture

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1 a high level of a melamine derivative, an unusually
high level of a flame-retardant and a high level of
hydrated alumina (also known as alumina trihydrate,
aluminium hydroxide, aluminium hydrate and hydrated
5 aluminium oxide).

Examples of melamine derivatives within the scope
of the formula given above are tris-(hydroxymethyl)
melamine, tris-(hydroxymethyl)-tris-(methoxymethyl)
melamine, hexa-(methoxymethyl) melamine, hexa-
10 (hydroxymethyl) melamine and tetra-(n-butoxymethyl)
melamine. In general, the amount of compound or com-
pounds of the formula used will be from about 10 to
about 30 parts by weight per 100 parts by weight of
the polyester polyol present in the mixture to be
15 foamed, but greater or lesser amounts can be used
without departing from the scope of the invention.
The preferred amount is 15 to 25 parts by weight, per
100 parts by weight of the polyester polyol (a). The
melamine derivatives can be used either as solutions
20 or dispersions in water or other solvents or as
essentially 100% active materials without solvent. It
is preferred to use the latter form, in the absence
of water, in order to provide greater latitude in
formulating a reaction mixture. When water is used
25 as a blowing agent, it is preferable to add it
separately, rather than as a solvent for the melamine
compound. A particularly preferred melamine derivative
is hexa(methoxymethyl)melamine, which is readily
obtainable in a form which is free, or substantially
30 free, from water. Although it is preferable to use a
single melamine derivative, for simplicity in formu-
lating, it may sometimes be desirable to use two or more
and such combinations are included in the scope of the
invention.

35 Examples of suitable flame-retardants are tris(1,3-

1 dichloropropyl) phosphate, tris(2,3-dibromopropyl)
phosphate, 2,2-bis(chloromethyl)-1,3-propylene-bis-
2 [di-(2-chloroethyl) phosphate], tris(2-chloroethyl)
phosphate, tris(2-chloropropyl) phosphate, bis(dichloro-
5 propyl)-tribromoneopentyl phosphate, tetrakis(2-
chloroethyl)-ethylene diphosphate (sold by Olin
Chemicals under the Regd. Trademark THERMOLIN 101),
oligomeric chloroalkyl phosphate products e.g. the
product sold by Stauffer Chemical Co. under the Regd.
10 Trademark FYROL EFF), tricresyl phosphate, cresyl
diphenyl phosphate, chlorinated paraffin and brominated
paraffin. Halogenated phosphates are preferred flame-
retardants in the practice of this invention, especially
tris(1,3-dichloropropyl) phosphate, tris(2-chloroethyl)
15 phosphate, the above-mentioned product FYROL EFF and
tetrakis(2-chloroethyl)-ethylene diphosphate, the first
and last-named being particularly preferred. Although
a single flame-retardant is preferable from the stand-
point of simplicity of formulation, mixtures of two or
20 more of the same type or of different types may be found
to give improved performance in some cases and such
mixtures are included in the scope of this invention.
The amount of flame-retardant can be varied over a wide
range, from 20 to 60 parts by weight per 100 parts by
25 weight of polyester polyol (a) in the reaction mixture.
It is preferable to use 20 to 40 parts by weight of
flame-retardant.

The amount of hydrated alumina incorporated can
range from 50 to 200 parts by weight per 100 parts by
30 weight of polyester polyol (a) in the foam-forming
reaction mixture, the preferred amount being in the
range from 90 to 120 parts by weight. Although the
particle size of the hydrated alumina is not critical,
too coarse a particle size may be ineffective in
35 producing a suitably intumescent foam and too fine a

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1 particle size may produce too high a viscosity and
thus cause difficulties in blending the reaction
mixture. Grades of hydrated alumina having an average
particle size of 16 microns or less are generally
5 not desirable, since they tend to produce too high a
viscosity, and grades containing coarse particles
(for example, in which 5% - 20% is retained on a 100-
mesh screen, 65% - 90% is retained on a 200-mesh screen
and 90% - 98% is retained on a 325-mesh screen) may
10 lead to some shrinkage of the foam. A suitable grade of
hydrated alumina, for example, is one in which 1% - 3%
is retained on a 200-mesh screen and 80% - 85% is
retained on a 325-mesh screen and which has an average
particle size of 20 - 30 microns. Some commercially-
15 available grades which are satisfactory for use in the
process and products of this invention are Alcoa C-30BF,
Imco 753, Southern Talc 753, Reynolds RH 130 and Alcoa
C-230.

Previously, it has been found very difficult or
20 virtually impossible to incorporate high levels of
fillers such as hydrated alumina into polyester poly-
urethane foam, especially when the foam is made by the
one-shot method. Polyester polyols are generally of
high viscosity and attempts to disperse large quantities
25 of particulate fillers in such polyols have given
mixtures of such viscosities that they were difficult
or impossible to pump or even to handle in any manner,
on a practical commercial scale. It has now been found
that, by using certain viscosity modifiers or synergists,
30 relatively low viscosity and pumpable dispersions of
hydrated alumina can be made. These dispersions are
readily handled and pumped and so are suitable for use
in the one-shot foam-making method. The dispersions
and the method of making them constitute important
35 preferred features of this invention.

1 In order to obtain a uniform distribution of hydrated
alumina throughout the reaction mixture and the
resultant foam, it is highly desirable to form a
premix of the hydrated alumina in all or part of one
5 or more of the liquid components of the reaction
mixture. Any of the known types of apparatus for
dispersing particulate solids in liquids can be used.

Hydrated alumina can be uniformly dispersed in the
high viscosity polyester polyol conventionally used
10 in the manufacture of polyurethane foam, such polyester
polyols typically having a viscosity in the vicinity
of 20,000 cps at 25°C, but the resultant dispersions
have extremely high viscosities, especially when the
amount of hydrated alumina is greater than about 50-75
15 parts by weight per 100 parts by weight of polyester
(50-75 PHR). Such dispersions can be used, although
with some difficulty, in making hand batches in the
laboratory, but are impractical for use in the apparatus
usually used for making foam by the continuous one-shot
20 process. Special pumps must be employed in order to
handle these high-viscosity dispersions in otherwise
conventional equipment.

It has now been found that dispersions of much lower
viscosity can be made by dispersing the hydrated alumina
25 in a blend of all of the polyester polyol, the flame-
retardant (either a single flame-retardant or a mixture
of two or more) and the melamine derivative. A preferred
technique in carrying out the process of the invention
is thus given, if the hydrated alumina (h), the or each
30 polyester polyol (a), the or each melamine derivative
(f) and the or each flame-retardant (g) are first
blended into a uniform dispersion before being combined
with the other components of the reaction mixture. The
resultant dispersions can be used in conventional
35 continuous foam-making equipment, without requiring

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1 modification such as the installation of special pumps.

It has also now been found that dispersions of even lower viscosity in some cases as low as 17,000-25,000 cps, can be made by dispersing the hydrated alumina in a blend which comprises all of the flame-retardant (or retardants if two or more are used), the melamine derivative and a low-viscosity linear polyester polyol. A further preferred technique thus involves the use of a reaction mixture which also comprises 5 to 20 parts by weight of (j), at least one second polyester polyol, per 100 parts by weight of the polyester polyol (a), the or each second polyester polyol (j) comprising a linear polyester prepared from a glycol and an aliphatic dibasic acid and having a viscosity of 400 to 500 cps at 25°C and wherein the hydrated alumina (h), the or each second polyester polyol (j), the or each melamine derivative (f) and the or each flame-retardant (g) are first blended into a uniform dispersion before being combined with the other components of the reaction mixture. These uniform dispersions are readily pumpable and can be introduced as a separate stream to the mixing head of a conventional apparatus for the continuous one-shot manufacture of polyurethane foam. They are not only lower in viscosity, which itself is an advantage, but they are also more convenient for use in the commercial manufacture of foam, since it is no longer necessary to handle large volumes of high viscosity polyester polyol in order to disperse the hydrated alumina. The process wherein such dispersions are made and used in foam making is a particularly preferred embodiment of the present invention.

The low-viscosity linear (i.e. difunctional) polyester polyols preferably used as such second polyols, in preparing the dispersions, can be made by

1 the reaction of a glycol, e.g. diethylene glycol,
with an aliphatic dibasic acid, e.g. adipic acid, and
typically have viscosities in the range from 400-500
cps at 25°C. Examples of suitable polyols are the
5 commercial products known as LEXOREZ 1100-220, sold
by Inolex Corp., and L-1217 and FOMREZ 11-225, both
sold by Witco. The amount of such low-viscosity linear
polyester polyol desirably used depends upon the amount
of hydrated alumina to be dispersed, the amount and
10 type of flame-retardant or retardants used, the amount
and structure of the melamine derivative and the
viscosity desired in the dispersion. It is preferable
to use 5 to 20 parts by weight per 100 parts by weight
of high-viscosity polyester polyol (a) (i.e. from 5 to
15 20 PHR), although greater or lesser amounts can be used
if desired without departing from the scope of the
invention. It is particularly preferable to use 10 to
15 PHR. The resulting dispersions thus comprise 5 to
20 parts by weight of low-viscosity linear polyester
20 polyol, 20 to 60 parts by weight of at least one halo-
genated phosphate ester flame-retardant, 10 to 30 parts
by weight of melamine derivative and 50 to 200 parts by
weight of hydrated alumina.

The low-viscosity linear polyester polyol not only
25 functions as a dispersant for the hydrated alumina, but
surprisingly it also enhances the flame-retardant and
intumescent properties of the resultant foam, as shown
by a highly significant improvement in Flame Spread
Index when tested by the method of ASTM E-162. This is
30 a completely unexpected result, since linear polyester
polyols have no inherent resistance to burning and
would be expected to be totally consumed by fire.

The mixture to be foamed includes (a) at least one
polyester polyol, (b) at least one organic polyisocyanate,
35 (c) at least one blowing agent, (d) at least one

1 surfactant and (e) at least one catalyst. The mixture
can also comprise other known additives and compounding
ingredients for the preparation of polyurethane foams.
Such additives and ingredients include, by way of
5 example, pigments or other colourants, foam stabilizers,
cell openers, lubricants, microbicides and so forth.

The polyester polyol reactants useful in carrying
out the present invention include any conventionally
used in the preparation of flexible and semi-flexible
10 urethane polymer foams. The polyhydric polyester
reactant usually has a molecular weight of at least 400
and optimally from 500 to 5000. The hydroxyl
number of the compound is correspondingly in the range
from 15 to 300. The preferred average hydroxyl function-
15 ality for the polyester resins is 2.2 to 2.8.

The polyester polyol compounds useful for preparing
the flexible polyurethane foams in accordance with the
present invention are well known and can be prepared,
for example, by the reaction of a polyhydric alcohol
20 with a polycarboxylic acid, each containing 2 to 36
or more carbon atoms in the molecule. The polycarboxy-
lic acid component can include such acid precursors as
the corresponding acid anhydrides or acid halides or
even, for example, the alkyl esters. The preferred
25 acids are dicarboxylic acids containing from 4 to 36
carbon atoms in the molecule. Examples of such
preferred carboxylic acid compounds include aromatic
acids, such as phthalic, terephthalic and tetra-
chlorophthalic acids, cycloaliphatic acids, such as
30 dimerized linoleic acid, maleated and fumarated resin
acids and cyclohexane-1,4-diacetic acid, and, especially,
aliphatic acids, such as itaconic, oxydipropionic,
succinic, glutaric, adipic, azelaic, suberic and sebacic
acids, or combinations of such acids. The polyester
35 polyols can also be prepared from the corresponding

1 lactones, for example, gamma-butyrolactone or epsilon-
caprolactone, by self-condensation on a diol or triol
initiator.

5 The polyhydric alcohol used in the preparation of
the polyester polyol preferably is, in general, a
mixture of a dihydric and a trihydric alcohol. Pre-
ferably, a mixture of polyols, the major portion having
a functionality of two and the minor a functionality
of three, is used. This mixture of di- and tri-
10 functional polyols is utilized to give an average
functionality between two and three. A functionality
greater than two is desirable, to provide cross-linking
in the reaction between the polyester polyol and the
polyisocyanate to form a flexible, but strong foam. It
15 has been found to be preferable to obtain this addition-
al functionality by using trihydric or higher polyols
in a minor amount when forming the polyester polyol.

It is recognized that certain compounds which are
considered by those skilled in the art to be polyester
20 resins also contain ether linkages, e.g., esters
prepared from dipropylene glycol. However, the primary
character of such resins is considered to be that of
an ester.

The organic polyisocyanates useful in carrying out
25 the present invention are also conventional. They
contain at least two isocyanate groups per molecule;
preferably, the isocyanate mixture selected has an iso-
cyanate functionality of from 2.0 to 3.0. Especially
useful isocyanates include aromatic polyisocyanates
30 alone or admixed with aliphatic, cycloaliphatic or
heterocyclic polyisocyanates.

The aromatic diisocyanates are generally the least
expensive and most suitable polyisocyanates available.
Such aromatic diisocyanates, especially the tolylene
35 diisocyanate isomers, are used commercially in the
preparation of flexible foams by the one-shot continuous

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1 slab-stock process. However, for certain purposes,
other polyisocyanates, especially aliphatic, aralkyl
and cycloalkyl polyisocyanates, have valuable properties
and can be used, if desired, in admixture with, e.g.
5 tolylene diisocyanates. The aralkyl, aliphatic and
cycloaliphatic polyisocyanates are especially useful
when resistance to degradation and discoloration due
to oxidation or light is needed. The non-aryl
polyisocyanates are generally not useful alone, but
10 can be used in combination with the other types for
special purposes.

Suitable organic polyisocyanates include, for
example, n-butylene diisocyanate, methylene diisocyanate,
m-xylyl diisocyanate, p-xylyl diisocyanate, cyclohexyl-1,-
15 4-diisocyanate, dicyclohexylmethane-4,4'-diisocyanate,
m-phenylene diisocyanate, p-phenylene diisocyanate,
3-(alpha-isocynatoethyl)-phenyl isocyanate, 2,6-
diethylbenzene-1,4-diisocyanate, diphenyldimethyl-
methane-4,4'-diisocyanate, ethylidene diisocyanate,
20 propylene-1,2-diisocyanate, cyclohexylene-1,2-
diisocyanate, 2,4-tolylene diisocyanate, 2,6-tolylene
diisocyanate, 3,3'-dimethyl-4,4'-biphenylene
diisocyanate, 3,3'-dimethoxyl-4,4'-biphenylene
diisocyanate, 3,3'-diphenyl-4,4'-biphenylene diisocya-
25 nate, 4,4'-biphenylene diisocyanate, 3,3'-dichloro-4,4'-
biphenylene diisocyanate, 1,5-naphthalene diisocyanate,
isophorone diisocyanate, m-xylene diisocyanate, triazine
triisocyanate, triketotrihydrotris(isocyanatophenyl)
triazine and tris(isocyanatophenyl) methane. The
30 preferred isocyanates used in the practice of this
invention are the tolylene diisocyanates, especially
mixtures of the 2,4 and 2,6 isomers such as those
containing by weight 65% of the 2,4 isomer and 35% of
the 2,6 isomer (TDI 65/35), and those containing
35 80% of the 2,4 isomer and 20% of the 2,6 isomer (TDI

1 80/20). In the manufacture of conventional polyester
polyurethane foams, the more expensive TDI 65/35 is
often used, because the reaction mixture tends to rise
and gel faster and the resultant foam tends to have
5 a more open cell structure. Surprisingly, in the
practice of this invention, the less expensive TDI
80/20 gives entirely satisfactory rise and gel times,
as well as a satisfactory degree of open cell forma-
tion, and is a particularly preferred isocyanate.

10 Generally, in carrying out urethane polymerization
reactions, the only significant groups in the reactant
compounds are the isocyanate groups and active hydrogen
groups which are reactive therewith. Acyclic, ali-
cyclic, aromatic and heterocyclic radicals are all
15 possible substituents on the active hydrogen and poly-
isocyanate reactants.

The preferred blowing agent for general use in the
production of flexible polyurethane foams is water.
The advantages of using water are low cost and the
20 stability which the use of water adds to the foam-
making. The water-isocyanate reaction not only
produces gas for blowing, but also produces urea-
containing polymer very quickly, contributing materially
to the early polymer strength which is needed to hold
25 in the gas and thus form foam. The water-isocyanate
reaction also provides much of the heat required for
satisfactory curing of the foam. Generally, when
water is used, it is present in proportions of 0.5
to 6 weight percent, based on the total weight of the
30 reacting polyols. Blowing agents which do not react
with the isocyanate can be used, either in conjunction
with water or as a total replacement for water. These
include compounds which are vaporized at the tempera-
tures produced by the exotherm of the isocyanate-
35 reactive hydrogen reaction. The various blowing agents

1 are well known in the art. Other known blowing agents
which are preferably used include certain halogen-
substituted aliphatic or cyclo-aliphatic hydrocarbons
having boiling points from -40° to $+100^{\circ}\text{C}$, including
5 methylene chloride, volatile fluorocarbons, such as
trichloromonofluoromethane, dichlorodifluoromethane and
1-chloro-2-fluoroethane and low-boiling hydrocarbons,
such as n-propane, cyclopropane, butane, isobutane,
pentane, hexane, cyclohexane and their mixtures.

10 As is conventional in the manufacture of poly-
urethane foam, the reaction mixture also contains a
catalyst. Catalysts suitable for use in this invention
are conventional ones in the polyurethane foam art and
include tertiary amines and metallic compounds. Useful
15 tertiary amines include N-alkylmorpholines, e.g. N-
ethylmorpholine, N,N-dialkylcyclohexylamines where the
alkyl groups are methyl, ethyl, propyl, butyl, etc.,
trialkylamines, e.g. triethylamine, tripropylamine,
tributylamine, triamylamine and so on, triethylenediamine,
20 bis(2-dimethylaminoethyl) ether, N,N-dimethylaminoethyl-
N',N'-dimethylaminopropyl ether, the tertiary amine
sold by Jefferson Chemical Co under the name THANCAT DD
and other tertiary amines well known in the art.
Useful metal compounds include those of bismuth, lead,
25 titanium, iron, antimony, uranium, cadmium, cobalt,
aluminium, mercury, zinc, nickel, cerium, vanadium,
copper, manganese, zirconium and tin. Tin compounds
are particularly useful, examples being stannous
octoate, (stannous 2-ethylhexoate), stannous oleate,
30 dibutyltin bis(2-ethylhexoate) and dibutyltin dilaurate.
The levels of catalyst used are conventional.

As is also conventional in the manufacture of poly-
urethane foam, the reaction mixture contains a
surfactant (if desired). The surfactant can be an
35 organosilicon surfactant (also commonly termed a

1 silicon surfactant) or it can be a surfactant which
does not contain silicon. It is generally preferred,
in carrying out this invention, to use the latter type,
although both types can be used, in combination if
5 desired. Regardless of the type used, either a single
surfactant or a combination of two or more can be used.
The surfactant per se does not form a part of this
invention. As those skilled in the art will be aware,
surfactants for use in making urethane foams are sold
10 by many suppliers, generally as proprietary products
without disclosure of exact chemical composition or
structure. The amounts used vary from 0.01 to 1 part
or more, per 100 parts by weight of the polyol (0.01
to 1 PHR). One such commercial non-silicon containing
15 surfactant suitable in carrying out this invention is
sold by Witco as M66-82 and is described as a mixture
of fatty acid amides.

In carrying out this invention, the index, namely
the stoichiometric balance between the equivalent
20 weights of the polyisocyanate and the total of polyol
and water (if used), can vary from a negative index
(less than 100, representing a shortage of polyiso-
cyanate) to a positive index (greater than 100,
representing an excess of polyisocyanate). The index
25 generally ranges from 95 to 105, but even lower or
higher indices can be used if desired. It has been
found that reaction mixtures having an index of at least
100 yield foams having better flame-retardance and
intumescent properties than those having an index of
30 less than 100. Thus an index of at least 100 is
preferred.

The polyurethane foams according to this invention
can be prepared by any of the methods known in the
art, including prepolymer, quasi-prepolymer and one-
35 shot, batchwise or continuous. It is preferable to

1 prepare them continuously by the one-shot method in
the form of large buns having a rectangular or
circular cross-section, which can then be split or
peeled into sheets of suitable thickness. For example,
5 buns having a circular cross-section and suitable for
peeling are disclosed in U.S. Patent Specification
3,874,988. The polyurethane foams can be essentially
completely open-celled, such as those disclosed in U.S.
Patent Specifications 3,748,288 and 3,884,848, or they
10 can be reticulated, by being made by any of the
reticulation methods known in the art. The foams can
also be partly open-celled, or predominantly closed-
celled.

One of the following examples is a comparative one
15 according to the prior art and the others, which are
according to the present invention, are illustrative,
but not limitative; in all the examples, all amounts
are given as parts by weight.

Test methods used for evaluating the flame-
20 retardant and intumescent properties of the foams
include a modification of UL 94, Standard for Tests
for Flammability of Plastic Materials for Parts in
Devices and Appliances, published by Underwriters
Laboratories Inc., ASTM E162-78, Radiant Panel Test,
25 the procedure of Technical Information Bulletin 117,
"Requirements, Test Procedure and Apparatus for Testing
the Flame Retardance of Resilient Filling Materials
Used in Upholstered Furniture" (referred to herein as
Calif. 117), published by the State of California,
30 Department of Consumer Affairs, Bureau of Home
Furnishings, the Michigan Mattress Test, and exposure
to a blowtorch.

The modification of UL 94 consists of starting to
count flame time from ignition, whereas in the standard
35 procedure flame time is counted 60 seconds after the

- 20 -

1 start of ignition.

The Michigan Mattress Test consists of rolling up a mattress, tying it either in the centre or at both ends, stuffing newspaper in the centre core of the
5 rolled-up mattress, placing the mattress at an angle and igniting the paper. The mattress is either totally consumed by the fire or the fire will self-extinguish after a time, the mattress being only partly damaged or consumed.

10 Exposure to a blowtorch is performed by suspending a strip of foam vertically and applying the flame of a propane blowtorch to the centre of the foam piece for a time ranging from 10 seconds to 3 minutes. At the end of that time, the torch flame is removed
15 and a visual assessment of the damage to the foam piece is made.

The following are the identities of various ingredients used in the examples. TDI 67/33 is a mixture of 2,4-tolylene diisocyanate and 2,6-tolylene diisocyanate in a 67/33 ratio. FYROL FR-2 is tris(1,3-dichloropropyl) phosphate and FYROL CEF is tris(2-chloroethyl) phosphate, both sold by Stauffer. NCM
20 is N-cocomorpholine. MM-70 is a mixture of 70% methoxyethylmorpholine and 30% dimethylpiperazine, sold by Texaco. C-4 is a 50% solution of stannous octoate in di(2-ethylhexyl)phthalate, sold by Witco.

Examples 1 to 14 are hand batches, prepared in the laboratory.

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35

<u>Example</u>	<u>Examples 1-5</u>				
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
Polyester *	100.0	100.0	100.0	100.0	100.0
TDI 80/20	30.0	30.0	30.0	33.2	46.5
Black pigment dispersion	6.0	6.0	--	--	--
Water	0.67	0.67	2.17	0.67	0.67
M66-82	1.0	1.0	1.0	1.0	1.0
NCM	1.85	1.85	1.5	1.5	1.5
C-4	0.50	0.20	0.15	0.15	0.20
Tris(methoxymethyl)-tris(hydroxymethyl)-melamine, 70% in water	5.0	5.0	--	10.0	15.0
Hexa(methoxymethyl)-melamine	--	--	20.0	10.0	--
FYROL FR-2	20.0	20.0	20.0	10.0	10.0
THERMOLIN 101	--	--	--	10.0	10.0
Hydrated alumina, IMCO 753	50.0	75.0	75.0	75.0	75.0
Cream time, sec.	10	15	20	15	10
Rise time, sec.	120	180	240	180	120

* Prepared from diethylene glycol, adipic acid and trimethylol ethane; OH no. 56

<u>Example</u>	<u>Examples 6-9</u>			
	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>
Polyester *	100.0	100.0	100.0	100.0
TDI 80/20	30.0	30.0	30.0	30.0
Water	2.2	2.2	2.2	2.2
M66-82	1.0	1.0	1.0	1.0
NCM	0.5	0.5	0.5	0.5
MM-70	0.75	0.75	0.75	0.75
C-4	0.15	0.15	0.15	0.15
Hexa(methoxymethyl) melamine	10.0	20.0	10.0	20.0
FYROL FR-2	20.0	20.0	20.0	20.0

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<u>Example</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>
Hydrated alumina, IMCO 753	50.0	50.0	75.0	75.0
Index	103	103	103	103
Cream time, sec.	15	15	15	15
Rise time, sec.	210	220	180	190
Density, lb/cu.ft.	5.42	5.37	5.65	5.98
Density, Kg/m ³	86.6	85.9	90.4	95.6
<u>UL 94-Unaged</u>				
Burn time, sec.	70.2	68.1	70.0	77.0
Burn extent, in.	1.10	1.02	1.00	0.97
Burn extent, mm.	27	25.7	25.4	24.6

- * Prepared from diethylene glycol, adipic acid and trimethylol ethane; OH no. 56

The foams from Examples 1-9 all had good intumescent properties, although those from Examples 1 and 2, which had a low level of substituted melamine as a char former, were noticeably inferior in intumescence to the others. Attempts were made to pre-disperse the hydrated alumina in a blend of the substituted melamine and the flame-retardant, but the viscosity of the dispersion was so high that it was necessary to add a portion of the polyester polyol to it.

Examples 10-12

<u>Example</u>	<u>10</u>	<u>11</u>	<u>12</u>
Polyester * (as above)	100.0	100.0	100.0
TDI 80/20	30.0	30.0	31.0
Water	2.2	2.2	2.2
M66-82	1.0	1.0	0.75
NCM	0.75	0.5	1.0
MM-70	0.75	0.75	0.5
C-4	0.15	0.15	0.15
Hexa(methoxymethyl)melamine	20.0	20.0	20.0
L-1217	--	--	10.0
FYROL EFF	40.0	--	--

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<u>Example</u>	<u>10</u>	<u>11</u>	<u>12</u>
FYROL FR-2	--	20.0	20.0
THERMOLIN 101	--	10.0	10.0
Hydrated alumina, IMCO 753	75.0	75.0	75.0

In order to alleviate the problem of high viscosity, higher levels of flame-retardants were used, together with the substituted melamine to pre-disperse the hydrated alumina in Examples 10 and 11; in Example 12, a low viscosity linear polyester polyol (L-1217) was also added to the dispersion. All gave viscosities which were low enough to be handled as a separate stream, without the necessity for adding any of the high viscosity polyester. The dispersion of Example 12 had the lowest and most satisfactory viscosity. The foams produced in each of these examples had excellent intumescence.

Examples 13-14

<u>Example</u>	<u>13</u>	<u>14</u>
Polyester * (as above)	100.0	100.0
TDI 80/20	31.0	35.0
Water	2.2	2.6
M66-82	0.5	0.5
NCM	1.25	1.25
MM-70	0.4	0.4
C-4	0.15	0.3
Hexa(methoxymethyl)melamine	20.0	20.0
FYROL CEF	10.0	35.0
FYROL FR-2	40.0	25.0
Hydrated alumina	90.0	110.0
Index	105	105
Cream time, sec.	10	10
Rise time, sec.	270	300

These examples illustrate the use of higher levels of hydrated alumina, added as a dispersion in a blend of the substituted melamine and the flame-retardants. The

dispersions were low enough in viscosity, without dilution with the polyester, to be handled by hand-batch techniques, but the viscosity was not as low as that of similar dispersions which also contained a low viscosity linear polyester. It was judged to be too high for optimum use as a separate stream in a one-shot foam-making machine. The foams from these examples had excellent intumescent properties.

Example 15

This is a comparative example, from a plant-scale production run, made according to the disclosure of U.S. Patent Specification 4,139,501. The reaction mixture contained amounts of melamine derivative and flame-retardant lower than those of the present invention and also did not contain hydrated alumina. The formulation was as follows.

Polyester * (as above)	100.0
TDI 67/33	30.0
Tris(methoxymethyl)tris(hydroxymethyl) melamine, 70% in water	5.0
FYROL FR-2	15.0
Water	0.67
NCM	1.0
M66-82	1.0
C-4	0.3
Pigment dispersion	6.0
Index	107
Density Kg/m ³ (= 3.70 lb/cu.ft)	59.3
Radiant panel flame spread index	
ASTM E162-78	550

Examples 16 to 23 were made on a pilot plant continuous one-shot foam-making machine using a conveyor declination of 1°, in the form of buns having a generally rectangular cross-section. In Examples 16 to 20, the buns had a width of approximately 86 cm (34 inches) while in Examples 21-23 the width was approximately

96 cm (38 inches).

In Examples 16 and 17, the hydrated alumina, polyester, hexa(methoxymethyl)melamine and FYROL FR-2 were blended into a uniform dispersion, which was introduced as a separate stream into the mixing head of the foam machine. A similar procedure was followed in Example 18, except that the THERMOLIN 101 was also incorporated into the dispersion. In Examples 19, 20, 22 and 23, the hydrated alumina, LEXOREZ 1100-220, hexa(methyloxymethyl)-melamine, FYROL FR-2 and THERMOLIN 101 were blended into a uniform dispersion, which was introduced as a separate stream into the mixing head. In Example 21, a similar procedure was followed, except that the dispersion did not contain THERMOLIN 101.

Examples 16-19

<u>Example</u>	<u>16</u>	<u>17</u>	<u>18</u>	<u>19</u>
Polyester * (as above)	100.0	100.0	100.0	100.0
TDI 80/20	29.0	31.0	29.5	31.0
Water	2.0	2.2	2.2	2.2
M66-82	1.0	1.0	1.0	0.75
NCM	1.0	1.0	1.0	1.0
MM-70	0.75	0.75	0.5	0.5
C-4	0.15	0.15	0.15	0.15
Hexa(methoxymethyl)melamine	20.0	20.0	20.0	20.0
FYROL FR-2	20.0	40.0	20.0	20.0
THERMOLIN 101	--	--	10.0	10.0
LEXOREZ 1100-220	--	--	--	10.0
Hydrated alumina	50.0	75.0	75.0	75.0
Index	105	105	100	95
Radiant panel flame spread index, ASTM E162-78	323	281	135	182

In addition to the radiant panel flame spread index, the properties summarized in Table I were measured.

Table I

<u>Example</u>	<u>16</u>	<u>17</u>	<u>18</u>	<u>19</u>
Density, lb/cu.ft.	4.61	6.38	5.86	5.87
Density Kg/m ³	73.7	104.0	93.8	93.9
ILD 50 mm (2") 25%	28.0	39.0	32.5	33.0
ILD 50 mm (2") 65%	64.0	89.0	74.0	90.0
Modulus 50 mm (2") 65/25	2.29	2.28	2.28	2.73
ILD 10 cm (4") 25%	38.0	54.0	43.0	44.0
ILD 10 cm (4") 65%	91.0	129.0	104.0	111.0
Modulus 10 cm (4") 65/25	2.39	2.39	2.42	2.52
Resilience, %	38.8	26.4	26.4	25.0
Tensile strength, lb/sq.in.	12.2	13.8	11.4	17.2
Tensile strength, Kg/cm ²	0.86	0.97	0.80	1.21
Elongation, %	190	170	160	190
Tear strength, lb/in.	2.0	2.3	2.1	2.9
Tear strength, Kg/cm	3.6	4.1	3.7	5.2
Flammability:				
UL 94-unaged	pass	fail	pass	fail
- aged.	--	fail	--	pass
Flammability:				
Calif. 117-unaged	fail	--	pass	pass
- aged	--	--	--	pass

Examples 20-23

<u>Example</u>	<u>20</u>	<u>21</u>	<u>22</u>	<u>23</u>
Polyester * (as above)	100.0	100.0	100.0	100.0
TDI 80/20	31.0	34.6	34.6	37.7
Water	2.2	2.2	2.2	2.5
M66-82	0.75	0.5	0.5	0.5
NCM	1.5	1.25	1.0	1.25
MM-70	0.4	0.4	0.4	0.4
C-4	0.3	0.15	0.15	0.15
Hexa(methoxymethyl)melamine	20.0	20.0	20.0	20.0
FYROL FR-2	20.0	40.0	20.0	20.0
THERMOLIN 101	10.0	--	10.0	10.0
LEXOREZ 1100-220	10.0	10.0	10.0	10.0

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<u>Example</u>	<u>20</u>	<u>21</u>	<u>22</u>	<u>23</u>
Hydrated alumina	90.0	90.0	90.0	90.0
Index	95	105	105	105
Radiant panel flame spread index, ASTM E 162-78	139	70	75	59

In addition to the radiant panel flame spread index, the properties summarized in Table II were measured.

Table II

<u>Example</u>	<u>20</u>	<u>21</u>	<u>22</u>	<u>23</u>
Density, lb/cu.ft.	6.02	6.87	6.09	5.21
" Kg/m ³	96.0	110.0	96.5	83.5
ILD 50 mm (2") 25%	33.5	48.0	42.0	28.5
ILD 50 mm (2") 65%	91.5	154.0	110.0	76.5
Modulus 50 mm (2") 65/25	2.73	3.21	2.62	2.68
ILD 10 cm (4") 25%	43.5	58.5	50.5	40.5
ILD 10 cm (4") 65%	120.0	162.0	135.0	104.0
Modulus 10 cm (4") 65/25	2.76	2.77	2.67	2.57
Resilience, %	25.0	33.3	3.19	29.2
Tensile strength, lb/sq.in	15.5	17.9	14.9	13.5
Tensile strength, Kg/cm ²	1.09	1.25	1.04	0.95
Elongation, %	170	130	140	150
Tear strength, lb/in.	2.7	2.5	2.7	2.4
Tear strength, Kg/cm	4.7	4.4	4.7	4.2
Flammability:				
UL 94-unaged	pass	fail	fail	fail
-aged	fail	--	--	--
Calif. 117-unaged	pass	pass	pass	pass
-aged	pass	pass	pass	pass

Comparison of the flame spread index of Examples 16-23 with that of comparative Example 15 illustrates the significant advantages over the prior art provided by this invention. The foams made according to the invention have outstanding resistance to burning under the intense ignition conditions of ASTM E162-78, even

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1 though some of them technically do not pass the less
rigorous test conditions of UL 94 and Calif. 117.
These foams may burn slowly on the surface and produce
a completely charred surface, while the interior of
5 the test piece remains unchanged, because of their
intumescent nature. The long burning time and apparent
extent of burn, which is in actuality only a surface
char, causes them to be rated as "failing" these less
severe tests.

10 Comparison of Examples 19 and 20 with Examples 22
and 23 with respect to ASTM E162-78 flame spread shows
the improvement which can be attained when the foam is
made at a positive index (105) rather than a negative
index (95).

15 Example 24

A mattress having dimensions 180 x 66 x 9cm
(72 x 26 x 3½ inches) and a density of 93.0 Kg/m³
(5.80 lb/cu.ft.) was prepared from the formulation
used in Example 18, and was subjected to the Michigan
20 Mattress Test. Within one minute after ignition of
the paper and flaming, there was some flaming of the
mattress itself with moderate smoke formation. Between
the second and sixth minutes, flaming of the mattress
continued, smoke generation diminishing from moderate
25 to low and with char formation on the mattress. After
seven to nine minutes, the fire appeared to be out and
smoke generation was very low. After ten minutes, the
mattress was unrolled. A heavy char layer was found on
the surface which had been in contact with the flaming
30 paper and along the top edge; the greater portion of
the mattress, the interior and the other surfaces
were undamaged.

Examples 25-27

Using the foams from Examples 21, 22 and 23,
35 mattresses having dimensions 180 x 90 x 7.5 cm (72 x

1 35 x 3 inches) were made and were subjected to the
Michigan Mattress Test. Each of these mattresses was
rolled up along its 90 cm axis around a 25 cm (10 inch)
diameter tube and tied together at each end. The tube
5 was removed and the rolled mattress was placed on the
ground outdoors, at an angle, with one end resting on
a 8cm (3 inch) angle iron to form a chimney with air
capable of entering at the bottom. Eight sheets of
newspaper 60 x 70 cm (23 x 28 inches) were then loosely
10 crushed into individual balls of about 15 cm (6 inches)
diameter, placed into the centre hollow of each mattress
roll and ignited from the top.

In Example 25 (foam from Example 21), during the
first four minutes after ignition, there was heavy
15 flaming from the paper with some flaming of the
adjacent foam surface, the formation of a heavy char
layer and moderate smoke generation. Between four and
nine minutes, the flames died down and went out at the
nine minute mark, although there was still some smoke
20 being generated. The mattress was unrolled and found to
have a heavy char layer on the surface which had been
contacted by the flame, about half of the total surface.
There was no smouldering of the unburned foam.

In Example 26 (foam from Example 22), during the
25 first four minutes there was heavy flaming of the paper
and some flaming of the foam, with moderate to heavy
smoke generation. Between five and twelve minutes,
the flames died down and smoke generation was moderate
to heavy. Some fire broke through to the outside of
30 the mattress where the roll was tied together with wire.
The flames had spontaneously extinguished at the fifteen
and one-quarter minute mark. When the mattress was
unrolled, there was somewhat more damage than in Example
25, but the greater part was not damaged. There was no
35 smouldering.

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1 In Example 27 (foam from Example 23), similar results
were obtained. There was heavy flaming and char forma-
tion during the first three minutes; the flames were
dying down during the next six minutes, with the forma-
5 tion of a very heavy char layer; and during the
following three minutes, the flames were flickering,
and then went out at the twelve minute mark. There was
a moderate amount of white smoke generated during the
last three minutes. The mattress was unrolled and
10 found to have a heavy char layer on the upper half, but
no other damage. There was no smouldering.

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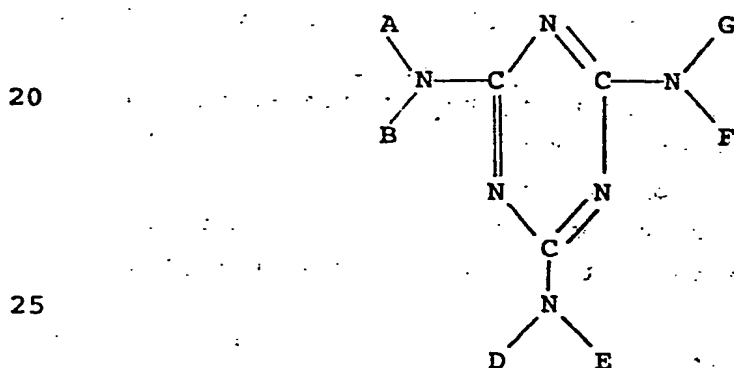
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1 : CLAIMS:

1. A process of manufacture of a flexible resilient flame-retardant and intumescent polyurethane foam from a reaction mixture comprising (a) at least one polyester polyol, (b) at least one organic polyisocyanate, (c) at least one blowing agent, (d) at least one surfactant, (e) at least one catalyst, (f) at least one melamine derivative and (g) at least one flame-retardant, wherein:

10 (1) the or each polyester polyol (a) comprises the reaction product of at least one polyhydric alcohol with at least one polycarboxylic acid or anhydride or at least one lactone, the polyester polyol having a hydroxyl functionality greater than 2 and a molecular weight of 400 to 5000;

(2) the or each melamine derivative, (f) has the formula:



wherein A, B, D, E, F and G are the same or different and each represents a hydrogen atom or a methylol or ROCH_2 group, in which R is an alkyl group having 1 to 4 carbon atoms, with the proviso that at least one of A, B, D, E, F and G is such a methylol or ROCH_2 group; and

(3) the flame-retardant is selected from triesters of phosphoric acid, halogenated triesters of phosphoric acid and halogenated hydrocarbons;

35 characterized in

1 that the amount of melamine derivative (f) comprises
10 to 30 parts by weight, per 100 parts by weight of
polyester polyol (a); that the amount of flame-
retardant (g) comprises 20 to 60 parts by weight, per
5 100 parts by weight of polyester polyol (a); and that
the reaction mixture also comprises 50 to 200 parts
by weight of (h) hydrated alumina, per 100 parts by
weight of polyester polyol (a).

2. A process according to claim 1, wherein the
10 amount of melamine derivative (f) is 15 to 25 parts
by weight, per 100 parts by weight of polyester polyol
(a).

3. A process according to claim 1 or 2, wherein
the melamine derivative (f) is hexa(methoxymethyl)-
15 melamine.

4. A process according to claim 1, 2 or 3, wherein
the amount of flame-retardant (g) is 30 to 40 parts by
weight, per 100 parts by weight of polyester polyol
(a).

20. 5. A process according to any preceding claim,
wherein the flame-retardant (g) is a halogenated
triester of phosphoric acid.

6. A process according to claim 5, wherein the
flame-retardant (g) is tris(1,3-dichloropropyl)
25 phosphate.

7. A process according to claim 5 or 6, wherein
the flame-retardant (g) is tetrakis(2-chloroethyl)-
ethylene diphosphate.

8. A process according to any preceding claim,
30 wherein the amount of hydrated alumina (h) is 90 to
120 parts by weight, per 100 parts by weight of
polyester polyol (a).

9. A process according to any preceding claim, when
carried out continuously by the one-shot method.

35 10. A process according to any preceding claim,

1 wherein the hydrated alumina (h), the or each polyester
polyol (a), the or each melamine derivative (f) and the
or each flame-retardant (g) are first blended into a
uniform dispersion before being combined with the other
5 components of the reaction mixture.

11. A process according to any preceding claim,
wherein the reaction mixture also comprises 5 to 20
parts by weight of (j), at least one second polyester
polyol, per 100 parts by weight of the polyester polyol, (a)
10 the or each second polyester polyol (j) comprising a
linear polyester prepared from a glycol and an aliphatic
dibasic acid and having a viscosity of 400 to 500 cps
at 25°C and wherein the hydrated alumina (h), the or
each second polyester polyol (j), the or each melamine
15 derivative (f) and the or each flame-retardant (g) are
first blended into a uniform dispersion before being
combined with the other components of the reaction
mixture.

12. A process according to claim 11, wherein the amount
20 of second polyester polyol (j) is 10 to 15 parts by
weight, per 100 parts by weight of polyester polyol (a).

13. A process according to any preceding claim,
wherein the index of the reaction mixture is at least
100.

25 14. A process according to any preceding claim,
wherein the polyisocyanate (b) comprises a mixture
of 2,4-tolylene diisocyanate and 2,6-tolylene diisocya-
nate in a weight ratio of 80/20.

15. A flexible resilient flame-retardant and intu-
30 mescent polyurethane foam, when made by a process
according to any preceding claim.